CARBON-POLYMER COMPOSITES FROM NATURAL PRECURSORS

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Abstract

The development of the technology referred to as Cellulose Derived Composites (CDC) is described as it relates to the production of carbon-polymer composites. Results from production of porous carbonized wood monoliths are described and measurements of permeability presented.

Introduction

Natural polymeric structures, such as wood, have been shown to be useful as a precursor for the production of advanced materials [1-6]. Commercially available carbon-polymer composites are used in a number of applications including those in aerospace, automotive, sports equipment and decorative markets. In structural applications they are valued for great strength and stiffness combined with low density. In decorative applications they are valued for their unique fabric-like texture combined with a deep silvery-black luster.

Production of carbon-polymer composites by the CDC process begins with a monolithic object – wood – and converts it to an intermediate state through controlled pyrolysis. This state is one that consists of carbon fibers (wood fibers converted to carbon) in an arrangement determined by the grain structure of the precursor. This carbonized wood monolith is then converted a to carbon-polymer composite by resin infiltration. Once the resin is cured, final machining operations can be performed including polishing to create a high luster that gives a material that is exceptionally appealing to the eye.

Successful production of carbon-polymer composites from wood requires both a highly controlled carbonization step and infiltration step. Carbonization of samples with dimensions greater that a few inches demands careful control over furnace heating rates, sample size and moisture content, and sample stacking arrangement. Improper care with any of these factors can lead to cracked or badly distorted samples. The carbonized sample must then exhibit permeability such that a polymer can fill the void spaces of the now-carbon wood cells. Successful infiltration results in a composite of remarkable beauty that has the color of ebony and grain features of the precursor wood species.

The permeability of wood has been studied for quite some time and is of particular interest to the forest products industries involved with preservative infiltration [7, 8]. It is well known that each wood species has different cell structures and, thus, differing permeability. Permeability is a consequence of a grain structure having interconnected hollow cells and is most often greatest in the sap-wood. Some wood species are more permeable than others, and usually exhibit differences from tree to tree. Models have been constructed to determine the manner in which fluids are transported through these porous structures [9].

Gas permeability, k, can be determined using experimental methods that employ Darcy's Law for flow resistance. This can take the form of

$$k = \frac{VLP}{tA\Delta P\overline{P}} \qquad (1)$$

where V/t is the volume flow rate, L is the flow length, P is the pressure at which volume flow rate is measured, A is flow cross-section, $\overline{P}$ is the average pressure, and ΔP is the pressure drop across the sample [9]. The gas permeability is then used as a measure of how readily a fluid may flow through the structure. Data indicates that flow is greatest in the grain direction and least in the direction tangent to the growth rings [7]. Oaks can be particularly permeable as a result of long, large cross-section vessel elements that serve as a direct path for flow. Other wood species may have orders of magnitude less permeability as a consequence of different pore structures/pathways.

Permeability of carbonized wood has yet to be reported in the open literature. While the present interest is in how this relates to fluid infiltration, it also has value when utilizing porous carbonized wood monoliths for structural adsorbents [10]. The characteristics of these materials enable unique application using a renewable natural resource.

Experimental

Carbonized wood was produced using methods described previously [1]. In the data presented here, various heating rates in the range of 25^{o}C to 800^{o}C were used to evaluate the effect on carbonized wood properties. Samples converted to carbon were then machined to dimension using either a vertical mill or abrasive paper.

Permeability was measured using the apparatus is shown in Fig. 1. Test samples are placed in a rubber washer that is squeezed between two plates. This compression caused Poisson expansion of the rubber such that a seal around the test sample is achieved. A vacuum pump is used to draw air though this system using the appropriate tubing and fittings. The air is thereby forced to pass through the sample. The resulting pressure drop is measured ($P_2 - P_1$) while the flow rate is recorded. Several rotameters (flow meters) were available for flow measure to encompass the range of flows expected in the various samples. The parameters measured were used to calculate the specific permeability of the sample.

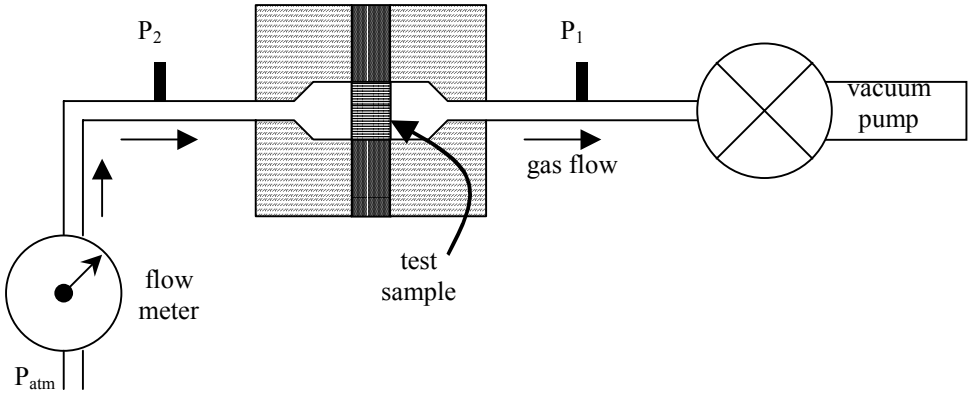

Figure 1. Schematic of permeability set-up for wood and carbonized wood.

Resin infiltration using epoxy resin/hardener was conducted as described in [3]. After resin cure, samples were shaped using either a vertical mill or abrasive paper, followed by polishing to a high luster with standard metallurgical methods. In some cases, clear coats were sprayed onto the surface to give depth of finish and added luster.

Results and Discussion

Carbonization studies have been used to refine both the product and efficiency of the process. Samples from furnace runs using similar heating sequences are shown in Fig. 2. These results illustrate the "pliable" nature of the wood as it is carbonized. Samples that were not weighted down (restrained from out-of-plane motion) during pyrolysis were warped and distorted in a manner similar to that when wood is allowed to dry without control over drying rate or without boundary restraint. Samples carbonized with boundary restraint were uniform in dimension and exhibited no warping. This was accomplished without the formation of cracks or macro-flaws.

Figure 2. Carbonized wood specimens processed without boundary restraint left, and with on right. Sample size of approximately 1.5 x 12 x 12 cm.

Heating rates have been studied to determine the optimum conditions for producing a carbonized wood sample. Altering heating rates influences carbon yield and dimensional changes. When heating rates increased by 25% in the temperature range of 200°C to 400°C the preliminary results shown in Table 1 were obtained. Faster heating resulted in lower yields and greater volume reduction.

	carbon yield		volume reduction	
	Poplar	Red Oak	Poplar	Red Oak
slow rates	28.01%	32.96%	63.88%	59.64%
high rates	24.72%	32.08%	63.94%	59.99%

Table 1. Influence of heating rate on yield and volume reduction.

The specific permeability of several species of carbonized wood was measured as shown in Table 2. Carbonized Red Oak exhibited the greatest permeability of all samples tested. Tupelo, a diffuse-porous hardwood of low density exhibited high permeability as well. Pine, Poplar and Redwood had the lowest permeability measured. These data, taken with flow in the direction of the grain (axial flow), compared somewhat to the values obtained from the precursor wood.

	Red Oak	Tupelo	Yellow pine	Yellow Poplar	Redwood
permeability	40.9	10.9	1.52	2.94	1.85

Table 2. Permeability of carbonized wood. Permeability is in units of in^4/lb sec. All of the data presented is an average of at least three tests of each specimen.

Samples of many species of carbonized wood have been thoroughly infiltrated with polymers to create a carbon-polymer composite. We have found that some species are more readily infiltrated, owing in part to differences in permeability. The end result is a carbon template filled with a polymer phase that gives improved mechanical properties. Composite samples are readily machined by conventional machine tools. When given a polish, the material exhibits a luster that highlights the grain features of the original wood. Such a sample is shown in Fig. 3.

Conclusions

This research has shown that the permeability of carbonized wood depends on sample species tested and gives low enough flow resistance to allow for resin transfer infiltration. The CDC process produces fully infiltrated carbon-polymer composites derived from wood and results in a unique material. While not all carbonized species infiltrate the same, our results are indicating that a wide variety of wood species can be used for this process. The permeability also demonstrates the capability for the

Figure 3. Carbon-polymer composite plate made from yellow pine.

carbonized wood to be used as a shaped structural filter or adsorbent medium. Further investigations are being conducted to demonstrate the market potential for this process which derives advanced materials from naturally renewable resources.

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